**Minor Nutrients in Fertilizers by AA or ICP**

Scope

This procedure is only suitable for the determination of metals in inorganic fertilizers.

Summary

The minerals in a well-ground portion of the sample are dissolved by heating with acid in a volumetric flask. After this treatment, the solution is brought to volume and mixed. This solution is then diluted, if necessary, to bring the element concentrations into atomic absorption standard curve ranges. The final dilution for calcium analysis must contain 1% lanthanum if air acetylene flame is used.

Comments

This procedure cannot be applied to fertilizers containing fritted trace elements. If zinc is to be determined, do not use rubber stoppers on the flasks. Samples prepared by this procedure can be screened by Inductively Coupled Plasma Spectroscopy.

Apparatus and Materials

A. 250 ml wide-mouth Pyrex volumetric flasks.

B. 15 ml disposable centrifuge tubes.

C. Hot plate.

D. Atomic Absorption Spectrophotometer.

E. Inductively Coupled Plasma Emission Spectrometer.

Reagents

A. HCl, concentrated.

B. 0.5N HCl: add 40 ml concentrated HCl to 920 ml deionized water and mix.

C. Lanthanum stock solution: 50 g La/L. Add 250 ml concentrated HCl to 58.65 g $La_2O_3$ in a 1 L volumetric flask. Add acid slowly as the reaction is violent and produces heat. Cool, dilute to volume with deionized water and mix.

Procedure

A. Sample preparation:

1. Weigh 1.00 g well ground sample (to the nearest 0.0001 g), into a 250 ml wide-mouth volumetric flask.

2. Add 10 ml concentrated HCl and boil gently 5-10 minutes on a hot plate in a well ventilated hood.

3. Cool and dilute to volume with deionized water.

4. Mix thoroughly and let stand so that particulates settle out.

B. Analysis by Atomic Absorption:

1. Dilute prepared sample solutions, if necessary, with 0.5 N HCl to bring element concentrations into atomic absorption standard curve range or ICP range.

2. Sample solutions for calcium analysis must contain 1% lanthanum if an air/acetylene flame is used. This can be accomplished by pipeting 20 ml of the final dilution into a 25 ml volumetric flask and diluting to volume with the lanthanum stock solution.

3. Consult the operator's manual for the instrument to be used. Parameters for each element are as follows for Perkin Elmer instruments:

| Element | Wavelength AA | ICP | AA Slit | AA Upper Limit ppm | Analyst's ICP Upper Limit |
|---|---|---|---|---|---|
| Calcium | 422.7 | 317.933 | 0.7 | 20 | 150 |
| Magnesium | 285.2 | 279.079 | 0.7 | 2.5 | 100 |
| Copper | 324.8 | 324.759 | 0.7 | | 30 |
| Iron | 248.3 | 238.203 | 0.2 | 20 | 100 |
| Manganese | 279.5 | 257.609 | 0.2 | | 50 |
| Zinc | 213.9 | 213.859 | 0.7 | 5 | 150 |

4. Read and record the absorbance values for each standard.

5. Calibrate in concentration mode using the appropriate calibration standards.

6. Read the concentration of the element of interest in each sample or have it printed out if a printer is available.

7. Run 10% checks and duplicates.

8. If a duplicate or check fails, recalibrate.

C. Screening by ICP:

1. Dilute any prepared sample solutions, if necessary, with 0.5 N HCl to bring element concentrations into ICP standard curve range.

2. Pour samples and standards into ICP autosampler tubes.

3. Set up 10% spikes and duplicates.

4. Refer to the ICP Operations Section of this manual to set up the instrument and analyze the samples.

5. Record the emission counts for each standard.

6. To have the ICP computer calculate results in %, divide the standard concentrations (ppm) by 10,000 before entering into the standards table. The sample weight and solution volume need to be entered in the sample analysis sequence list.

## Calculations

A. For atomic absorption results where element concentrations are determined as ug/ml, calculate the % of the element as follows:

$$\% \text{ element} = (\text{ug/ml in final diln})(F/\text{sample wt.})(10^{-4})$$

where:

$$F = \frac{\text{(ml original volume)(each intermediate volume)(final volume)}}{\text{(each intermediate aliquot)(final dilution aliquot)}}$$

B. The ICP or AA operator can calculate the spike recoveries. Consult QC charts to determine if recoveries are acceptable. If recoveries are not acceptable, the spike should be redone to determine if the entire run needs to be repeated.

C. The ICP results will be automatically calculated if the computer was set up to do so. The ICP operator can perform data reduction after the run and will be able to transfer satisfactory results from the ICP computer to the laboratory information management system.

Quality Control

A. Use $\geq$ 10 ml pipets and $\geq$25 ml volumetric flasks for any dilutions for AA analysis.

B. Document the instrument settings and standardization.

1. Record the adsorbance value or emission counts for each standard for each run. These should be on a printout.

2. Keep a record of wavelengths and analytical ranges used for each element. This is also on a printout.

C. Run a check and a duplicate for every ten samples. If difficulties with recovery occur,

1. Enter check recoveries into the computer QC system so that a standard deviation can be calculated.

2. Check recovery should be within two standard deviations of the mean recovery. If it is not, rerun the checks and adjust conditions until acceptable recovery is obtained and rerun the sample set if necessary.

3. Spikes can be used also in the run. It is useful to use them when running new calibration standards.

Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., secs. 2.126-2.130

JAOAC 50, 158-159 (1975)